Title: Perkin-Elmer Optima 2000 ICP OES General Use

Purpose:

The purpose of this SOP is to provide basic operating procedures for the Perkin-Elmer Optima 2000 ICP-OES system located in the Chemistry Laboratory at the Stuttgart National Aquaculture Research Center (SNARC).

Scope:

This procedure applies to basic start-up and shut-down of the instrument, it does not contain information on how to perform maintenance or troubleshooting. If problems arise while using the instrument, please notify the chemist and document the problem in detail on the instrument utilization record (System Use Form). The chemist and vendor specialists share responsibility for maintenance and repair of the Optima 2000 system. All maintenance performed by the chemist will be documented in the instrument notebook. Installation/Operational Qualification, Performance Verification, Preventative Maintenance and unscheduled maintenance performed by vendor specialists are certified and documented. This documentation is contained in the Instrument System log book located in the chemist's office.

It is the responsibility of each individual performing work in the Chemistry Laboratory to read all SOPs related to safety procedures for tasks the individual performs. SOPs should be reviewed prior to work commencement.

Health/Safety Warning:

When working with potentially hazardous materials, follow EPA, OSHA, and other specific health and safety procedures. Be prepared in case of emergency (e.g., telephone numbers, first aid kit). Personnel should wear an apron or lab coat, gloves and safety glasses when handling hazardous chemicals. Consult MSDS before handling chemicals.

User Responsibilities for ICP use:

User must be trained on the instrument by the chemist before being assigned a user ID and temporary password allowing access to the ICP's computer system.

User should provide an individual method for analysis. The chemist is available for help if needed; advanced notification is required.

User must keep record of analyses in the Instrument System Use log. This will include Date, Name, Project Leader, Element Analyzed, Standards Used/Amount, Matrix Composition, Total # of Samples Analyzed and a Detailed Explanation of Errors.

User must export data collected in a timely fashion to avoid loss of data, preferably after run completion. Do not attempt to do this during ICP analyses.

Users that require special configurations for analysis (i.e., changing nebulizers, spray chambers, using organic solvents, etc.) must inform the chemist one day prior to analysis. The chemist will configure the instrument; do not attempt to do this.

Equipment and Supplies:

Perkin-Elmer Optima 2000 ICP OES System
Nitric Acid - Trace Metal Grade (CAS # 7697-37-2; ex., Fisher Chemical # A509SK-212)
Milli-Q-water

Reagents:

Nitric acid solution, 1%: Fill a 2 L volumetric 2/3 full with Milli-Q water (see SOP#GEN 101.0). Nitric acid is located in a dispenser under the fume hood in the Acid Digestion Room #133A. Wearing proper safety glasses, lab coat and acid resistant gloves, measure 20 ml nitric acid into a graduated cylinder under the fume hood (see SOP#GEN 102.0). Working under the fume hood, pour the nitric acid into the volumetric. Bring to volume with Milli-Q. This 1% nitric acid solution is used as the wash solution.

Procedure:

1. Check gases; the liquid argon tank must be at least 25% full on the outside tank monitor and the gauge should read 80 psi; the air compressor gauge should read 80 psi.

2. Turn the chiller on by pressing the **On** button; the pressure should read 50-52 psi.

3. Verify that the Optima 2000 is **on**; the power switch is on the right side of the instrument. The instrument should be left on Monday AM through Friday PM.

4. Inspect the peristaltic pump tubing. The tubing is good for 8 hours of use; if the tubing is flattened in any place, replace the tubing.

5. Verify there is enough wash solution. The default solution is 1% Nitric Acid (Trace) in Milli-Q-water.

6. Verify that the waste container has enough room for waste generated during the sample run. If the 15 L waste container is full, contact the chemist for waste disposal.

7. Start the computer; see Chemist for system password. Log in with an assigned user ID and password. Start the WinLab32 (ver. 3.0.0.0103 ES) software on the computer by double clicking on the **WinLab32** icon. It will take several minutes for communication to be established and the optics to be initialized. (The WinLab32 software should be on for at least 2 hours). Select **Tools** on the menu bar followed by **Spectrometer Control**. In the **Manual Settings** portion of the window set **Purge Gas** to **Normal** and select **Apply**; an **Explanation for Action** window will appear and the reason for system modification must be entered, select **OK** and minimize the **Explanation for Action** window.

8. Move the autosampler probe to the wash location by selecting **Analysis** on the menu bar followed by **Autosampler** and then **Go to Wash**. The instrument will not rinse unless the probe is in the wash.

9. Ignite the plasma by clicking on the **Plasma** icon in the toolbar. Click on the **Plasma ON** switch in the **Plasma Control** window and wait until the plasma has ignited. A green glow will be evident in the observation window. Verify that the green lights are on each icon within the **Plasma Control** window EXCEPT for the **Heat** and **Flush** icon. The instrument takes 25 - 30 minutes to warm up before starting analysis.

10. Observe the autosampler peristaltic pump flow after the plasma has been ignited to ensure that the tubing has been properly installed; the sample tubing (black) is drawing sample and drain tubing (red) is draining sample. After verifying that the instrument is working properly, click on the **Pump** icon within the **Plasma Control** window to turn the instrument's peristaltic pump off. Remove the autosampler probe from the wash by selecting **Analysis** on the menu bar followed by **Autosampler** and then **AS Probe Up/Down**.

11. Create a method and sample information file. See below for instructions on how to do each of these.

12. Load the autosampler. The autosampler has tray sets allowing 62 or 157 numbered sample positions. The number that corresponds to each position is listed in the bottom left corner of each tray. The 62 sample tray holds 50 mL tubes. The 157 sample tray holds 50 mL tubes in the first 8 positions and 15 mL tubes in the remaining positions. Position zero of either set is the home position for the autosampler probe and is filled with a 1 - 5% HNO_3 wash solution. The 157 sample tray is the default autosampler setting; contact the chemist to choose the 62 sample tray setting. Make sure that the reservoir has sufficient wash solution in it prior to run, and that the autosampler is in the **On** position. Positions 1 – 157 can be used for either blanks, standards, QC samples or samples. Use polypropylene sample tubes and make sure that there is sufficient liquid in each to compensate for the flow rate vs. delay, flush, and read times.

13. Attach peristaltic pump tubing prior to analyses. After attaching tubing, turn the pump on by clicking the **Pump** icon within the **Plasma Control** window.

14. Make sure the specific method and sample info file is loaded in the main **WinLab32** screen. Start the analyses by clicking on the **MethEd** icon, then select **File** on the menu bar followed by **Print** and then **Active Window** to print a hard copy of the method to include with the final data. Minimize the method and click on the **Auto** icon in the toolbar to ensure the method and sample info files listed are correct in the **Setup** tab; this will open the **Automated Analysis Control** window. Enter the filename to save in **Results Data Set Name** and place a check in the **Save Data** box. Place a check in the **Print Log During Analysis** box as a precautionary measure. Click the **Analyze** tab; the sample schedule will appear containing the calibration blank, standards, and samples as defined in the method and sample info file. Click on **Print List** for a copy of the sample schedule and use it to ensure that samples are in their assigned positions. Click the **ANALYZE ALL** button. Verify that the calibration curve is acceptable, and that the instrument is working properly.

Creating a Method

Use the **Winlab Software32 Method Editor** in the **WinLab32** software by selecting **File** on the menu bar followed by **New** and then **Method**. In the **Create New Method** window, accept **Default** for **Starting Conditions** and **Aqueous** for **Plasma Conditions**. Click **OK**.

The method consists of several pages, and each page may contain multiple tabs. The pages are listed at the bottom of the window, and the tabs for each page are listed on the right. The pages and tabs will open with many default settings. Unless otherwise instructed, do not change the default settings. Follow the instructions below for each page and tab.

Spectrometer page:

***Define Elements tab*:** Type a method description. Enter elements and wavelengths of interest by clicking on **Periodic Table**. When the periodic table appears click on the element of interest. Click on the λ **Table**, a list of wavelengths for that element will appear and the recommended wavelength will be selected. Highlight the entire row and click **Enter Selected Wavelengths in Table**. Continue until all elements and wavelengths of interest are entered. Close open windows.

NOTE: The instrument measures wavelengths one at a time. Multiple wavelengths and elements will require more time and sample for analyses.

***Settings tab*:** Change only the following:

If sample volume allows, under **Read Parameters**; click on **Set** for Time (sec) and set **Min** to **5** and **Max** to **10**. Click **OK**.

Set **Replicates** to **3**.

Delay time: Use 60 sec for autosampler and 90 sec for manual analysis.

***Spectral Windows tab*:** Use default settings.

Sampler page:

***Plasma tab*:** Use default settings.

***Peristaltic Pump tab*:**

Note: Change the Sampler Flow Rate as above only if method requires slower rates for small sample amounts, if not use default settings.

Sample flow rate (mL/min) set at **1.0**.

Flush time (sec) set at **10**.

***Autosampler tab*:** Use default settings.

Process page:

***Peak Processing tab*:** Use default settings.

***Spectral Corrections tab*:** Use default settings.

***Internal Standards tab*:** Select **NO** for **Do Calib Blanks Contain Internal Standards?** (Unless a standard is added to each sample). Use default settings for all other options.

***Internal Standards Check tab*:** Use default settings.

Calibration page:

***Define Standards tab*:** Enter calibration blanks, standards, and their numerical locations in the autosampler.

***Calib Units and Concentrations tab*:** Each standard entered in the **Define Standards** tab will have a column here.

Choose the Calibration Units for each element by using the drop down menu next to the element. Enter the exact concentration of each element for a given standard. Or, highlight multiple boxes in a column by clicking and dragging with the mouse, then right click on the highlighted area, choose **Column Fill**, enter the value, and click **OK**.

Use default settings for all other tabs.

Checks page: Use default settings.

QC page:

QC sample Definition tab: Fill in **QC Sample ID:** for Initial and Continuing Calibration Verification and Blank samples.

Concentration and Limits tab: Select each QC sample in the **QC Sample ID** window and set upper and lower limits for run failure (usually 15% ± CONC).

Schedule QC tab: Place a check under ICV and ICB for **After Initial Calib** and CCV and CCB for **Periodically**. Under **Periodic Timing of Analysis**, select **Same for All QC's** and set to 10.

Use default settings for all other tabs.

Options page: Use default settings.

Saving a Method:

Save the method by selecting **File** on the menu bar followed by **Save As** and then **Method**. Enter a unique name and click **OK**.

This filename will appear as the current method at the top of the main **WinLab32 ICP Continuous** window.

NOTE: Default settings will take care of most analyses; adjustments may be needed for special applications or difficult samples.

Creating a Sample Information File:

Click on the **SamInfo** icon in the toolbar. A **Sample Information Editor** window will open; fill in a file description for all samples. Fill in the table with the **A/S Location** and **Sample ID** information for each sample, blank or standard. After standards are run, the autosampler will run the Sample Information File in the order of this table. Save the file by selecting **File** on the menu bar followed by **Save as** and then **Info File.** This filename will appear as the current Sample Info File next to **Sample Info** at the top of the main **WinLab32 ICP Continuous** window.

End of Run Procedure:

Flush the system with 1-5% HNO_3 for several minutes. The wash solution is 1% nitric by default; the autosampler probe must be moved to the wash location by selecting **Analysis** in the file menu followed by **Autosampler** and then **Go to Wash**. After flushing the instrument, remove the autosampler probe from the wash by clicking on **Analysis** in the file menu followed by **Autosampler – Probe Up/Down**.

Turn off the peristaltic pump by clicking on the **Pump** button within the **Plasma Control** window; manually release the tubing clamp and loosen both the sample line and drain line tubing from the peristaltic pump.

Select the **Plasma OFF** switch in the **Plasma Control** window, and wait until the plasma has been extinguished; there should no longer be a green glow through the observation window.

Shut Down:

Manually turn off the chiller by pressing the **Off** button.

Turn off the Optima 2000 system with the power switch on the right side of the instrument.

Remove all samples, solutions and clutter from the work space.

Calculations/Data Handling/Documentation:

Make sure the run has been recorded and all pertinent information is in the instrument System Use Log.

References:

APHA (American Public Health Association), American Water Works Association, and Water Pollution Control Federation. 1998. Standard Methods for the Examination of Water and Wastewater, 20th edition. Washington D.C.

Anonymous. 2001. Optima 2000 ICP Optical Emission Hardware Guide. PerkinElmer Instruments LLC, Shelton, CT.

Boss, C.B. and K.J. Fredeen. Concepts, Instrumentation and Techniques in Inductively Coupled Plasma Optical Emission Spectrometry, 3rd edition. PerkinElmer Life and Analytical Sciences, Shelton, CT.

USDA/ARS – Stuttgart/Pine Bluff Location – Safety Health and Security Plan.

USDA/ARS – SNARC – SOP# GEN 101.0: Deionized and Milli-Q Reagent Water Systems

USDA/ARS – SNARC – SOP# GEN 102.0: Fume Hood

Barker, K. 1998. At the Bench: A Laboratory Navigator. Cold Spring Harbor Laboratory Press. 460 pp.

Pat Malone — Prepared by — 9-14-2005 — Date

Dave Straus — Lab Supervisor — 9-14-2005 — Date